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Description

The present invention relates to enzymatic dishwashing or rinsing processes using compositions comprising lipolytic enzymes, and the manufacture of such compositions using ingredients specified herein.

5 EP-A-0 130 064 (Novo) concerns detergent additives based on Fusarium oxysporum lipase (e.g. 20,000-100,000 LU/g, used in detergent at e.g. 0.2-2% w/w.

DE-A-1 930 636 (= GB 1 273 545) (Albright & Wilson) concerns cleaning compositions based on protease, lipase and/or decarboxylase (of unspecified type) and surfactant, used inter alia for dishwashing.

EP-A-0 205 208 (Unilever) (not prepublished) discloses enzymatic detergent compositions comprising
10 lipase with 1-40% anionic surfactant and builders. The lipases disclosed are those that cross-react with antibody against lipase from Pseudomonas fluorescens 1AM 1057.

EP 0 206 930 (Unilever) (not prepublished) also discloses enzymatic detergent compositions comprising similar lipases together with combinations of anionic and nonionic detergent.

Thus, enzymatic dishwashing compositions have been proposed in the art. As enzymes, mainly
15 amylases and/or proteases have been proposed for inclusion in dishwashing compositions. Lipases have also been suggested, but have received far less attention than the amylases and/or proteases. Dishwashing compositions, in particular machine dishwashing compositions which are used in the main wash step of a machine dishwashing operation, have in general a satisfactory cleaning performance. However, frequently the article cleaned with such products still do not have a satisfactory visual appearance after rinsing and
20 drying, showing film or spots. Some main wash liquor is usually carried over from the main wash step to the rinse step, causing some deposition of soil resulting in insoluble calcium salts on the articles to be rinsed, which results in visible film or spots on the articles when they are dry. In particular with glass articles, this causes an unsightly visual appearance.

We have now found that the addition of lipases to a main wash dishwashing composition or to a rinse
25 composition significantly reduced the formation of film or spots on the articles cleaned or rinsed with such a composition.

Accordingly the invention provides a process for machine dishwashing or rinsing, comprising treating dishes with an aqueous liquid having dissolved or dispersed therein an effective amount of an enzymatic dishwashing or rinsing composition comprising from 0.5-10% by weight of a detergent surfactant and from
30 0-90% by weight of a builder, characterised in that it further comprises from 0.005-100 LU/mg of a lipolytic enzyme.

The lipases, used according to the present invention, may be of any suitable origin such as yeasts, fungi and bacteria. Preferably, however, they are of bacterial origin, and particularly preferably they belong to the class of bacterial lipases which show a positive immunological cross-reaction with the antibody of the
35 lipase, produced by the microorganism Chromobacter viscosum var lipolyticum NRRL B-3673.

This lipase has been described in Dutch Patent Specification 154,269 of Toyo Jozo, and the microorganism is available to the public at the United States Department of Agriculture, Agricultural Research Service, Northern Utilisation and Development Division at Peoria, Illinois, under the number NRRL B-3673. This lipase will hereinafter be referred to as "Toyo Jozo" lipase. The preferred lipases to be used
40 in the present invention should show a positive immunological cross-reaction with the Toyo Jozo lipase antibody, using the standard and well-known immunodiffusion procedure according to Ouchterlony (Acta. Med. Scan., 133, pages 76-79 (1950)).

The preparation of the antiserum is carried out as follows:

Equal volumes of 0.1 mg/ml antigen and of Freund's adjuvant (complete or incomplete) are mixed until an
45 emulsion is obtained. Two female rabbits are injected with 2 ml samples of the emulsion according to the following scheme:

day 0 : antigen in complete Freund's adjuvant
day 4 : antigen in complete Freund's adjuvant
day 32 : antigen in incomplete Freund's adjuvant
50 day 60 : booster of antigen in incomplete Freund's adjuvant.

The serum containing the required antibody is prepared by centrifugation of clotted blood, taken on day
67.

The titre of the anti-Toyo Jozo-lipase antiserum is determined by the inspection of precipitation of serial dilutions of antigen and antiserum according to the Ouchterlony procedure. A 2⁵ dilution of antiserum was
55 the dilution that still gave a visible precipitation with an antigen concentration of 0.1 mg/ml.

All lipases showing a positive immunological cross reaction with the Toyo Jozo lipase antibody as hereabove described are preferred lipases for use in the present invention. Typical examples thereof are the lipases ex Pseudomonas fluorescens IAM 1057 (available under the trade name Amano-P), the lipase ex

Pseudomonas fragi FERM P 1339 (available under the trade name Amano-B), lipase ex *Pseudomonas nitroreducens* var. *lipolyticum* FERM P 1338, the lipase ex *Pseudomonas* sp. available under the trade name Amano-CES, the lipase ex *Pseudomonas cepacia*, lipases ex *Chromobacter viscosum*, e.g. *Chromobacter viscosum* var. *lipolyticum* NRRLB 3673, commercially available from Toyo Jozo Co., Tagata, Japan; and further *Chromobacter viscosum* lipases from US Biochemical Corp., U.S.A. and Diosynth Co., The Netherlands, and lipases ex *Pseudomonas gladioli*.

The lipases used in the present invention are included in the detergent composition in such an amount that the final detergent composition has a lipolytic enzyme activity of from 100 to 0.005 LU/mg preferably 25 to 0.05 LU/mg of the composition.

A Lipase Unit (LU) is that amount of lipase which produces 1 μ mol of titratable fatty acid per minute in a pH stat. under the following conditions: temperature 30 °C; pH = 9.0; substrate is an emulsion of 3.3 wt.% of olive oil and 3.3% gum arabic, in the presence of 13 mmol/l Ca^{2+} and 20 mmol/l NaCl in 5 mmol/l Tris-buffer.

Naturally, mixtures of the above lipases can be used. The lipases can be used in their non-purified form, or in a purified form, e.g. purified with the aid of well-known adsorption methods, such as a phenylsepharose-adsorption techniques.

The composition of the invention may furthermore comprise the usual ingredients of dishwashing or rinse compositions. Thus it may contain one or more alkali salts commonly used in dishwashing compositions. Thus, it may contain organic and/or inorganic builders such as the alkali metal ortho-, pyro and tripolyphosphates and hexametaphosphates, silicates, carbonates, zeolites, borates, citrates, carboxymethyloxy-succinates, nitrilotriacetates and ethylenediamine-tetraacetates, polymeric polyelectrolytes such as polyacrylates, polymaleates, and other known organic and inorganic builder compounds.

Usually, the amount of builders in the composition varies from 10-90% by weight, generally from 30-70% by weight.

The composition may also contain a detergent-active compound. If a detergent-active compound is included, it usually is in an amount of from 0.5-10%, usually 1-5%. Any well-known type of detergent active compound may be used, such as soaps, synthetic anionic, non-ionic, amphoteric detergent surfactant and mixtures thereof. Preferably, a nonionic detergent surfactant is used, especially a low-foaming one. Suitable examples of such nonionic detergent surfactants can easily be found in M.Schick "Nonionic Surfactants" (1967).

The composition may furthermore contain other useful additives such as bleaching agents, bleaching agent activators, hydrotropes, fillers, perfumes, colouring agents, germicides, soil-suspending agents, aminopolymetaphosphonic acids and alkali metal or alkaline earth metal salts thereof, clays such as hectorites, anti-corrosion agents such as fatty acids, benzotriazole and so on. Other enzymes such as proteases, amylases, oxidases may also be included.

As bleaching agents the peroxygen type bleaching agents, preferably with a bleach precursor such as TAED are suitable for inclusion in the machine dishwashing compositions.

A typical example of a conventional machine dishwashing composition usually contains an alkali metal tripolyphosphate in an amount of from 20-60%, an alkali metal silicate in an amount of from 40-80%, or an alkali metal disilicate in an amount of 5-30% by weight a peroxy type bleaching agent in an amount of from 1-15%, a low-foaming detergent surfactant in an amount of from 0.5-5%, and minor ingredients such as perfumes, colouring agents, hydrotropes, fillers, etc.

When formulated as a rinse composition, it may contain from 0.5-10% by weight of a nonionic surfactant, from 5-25% by weight of an inorganic or organic acid, from 1-5% by weight of a hydrotrope, dyes, germicides, clays, silica sols, etc., the balance being water. The products of the invention can be formulated in any desirable form, such as powders, granulates, cakes, bars, pastes, liquids etc. The invention will further be illustrated by way of example.

Example 1

In a commercial dishwashing machine tests were carried out under the following conditions:

detergent composition dosage: 30 g

lipase dosage : 2.5 g (= 15 LU/ml)

5 water hardness : main wash 27° GH
flood 9° GH
rinse 1 9° GH
10 rinse 2 9° GH
final rinse 9° GH

wash temperature and time : 55°C - 60 minutes

articles to be cleaned : clean dishes
15 clean glasses

test soil : 35 g egg-yolk, added at
the beginning of the main
20 wash.

The detergent composition had the following formulation:

25		in gr.
	sodium tripolyphosphate	11.61
	sodium carbonate	2.7
	TAED (59.7%)	1.81
30	sodium perborate	2.43
	calcium-Dequest (47.6%)	0.397
	sodium disilicate	3.3
35	low foaming nonionic	0.405
	(Plurafac RA40)	
	hectorite clay (Laponite XLS)	0.675
	sodium sulphate	5.610
40	amylase (4.8 Maltose units/mg)	0.540

As lipase, the Toyo Jozo lipase was used.

45 The test was carried out in two runs, one without and one with lipase.

The glasses were appraised by an expert panel as to their visual appearance, and were given a score according to the following scale:

	<u>Film</u>	<u>Spots</u>
50	1. no film	1. no spots
	2. slight film	2. 1-5 spots
	3. clear film	3. 5-10 spots
55	4. heavy film	4. 11-20 spots
		5. more than 20 spots

The following results were obtained:

	run 1 (no lipase)	run 2 (with lipases)
5	average film score : 2.3	2.3
	average number of spots : 250	100

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Example 2

The test of Example 1 was repeated, but under the following different conditions:

15

wash temperature : 65°C

soil : standard soil; at the end of the
main wash, clean plates were put in
the machine, so that no cleaning
took place in the final rinse, but
only a carry-over took place.

20

run 1 : no lipase added in the final rinse

25

run 2 : 2.5 g Toyo Jozo lipase was added to
the final rinse (15 LU/ml).

30

The main wash composition had the following formulation:

	% by weight
35	sodium tripolyphosphate 36
	sodium carbonate 9
	sodium disilicate 15
	TAED 3.8
40	sodium perborate tetrahydrate 8.8
	potassium metaborate 0.35
	sodium metaborate 0.85
45	calcium-Dequest 0.63
	Amylase (4.8 MU/mg) 1.8
	Esperase (a protease; 675 GU/mg) 1.8
	low-foaming nonionic (Plurafac RA 40) 1.35
50	hectorite (Laponite XLS) 2.25
	benzotriazole 0.1
	sodium sulphate 14.0
55	water 9.5

The following spot and film scores of the glasses were obtained:

run 1 (no lipase)	run 2 (with lipases)
average film score : 1	1
average spot score : 5	2.7

Example 3

Replacing the Toyo Jozo lipase by the Amano-P lipase or Lipase MY (ex Candida cylindracea) or Amano AP-6 lipase (ex Aspergillus niger) in Example 2 gave similar results.

Example 4

The following formulation is an example of a rinse aid composition to which lipases can be added:

	% by weight
low-foaming nonionic (Plurafac RA 30)	10
citric acid	18
sodium xylene sulphonate	3.0
dye	0.003
butylated hydroxytoluene	0.1
water	q.s.

Claims

1. A process for machine dishwashing or rinsing, comprising treating dishes with an aqueous liquid having dissolved or dispersed therein an effective amount of an enzymatic dishwashing or rinsing composition comprising from 0.5-10% by weight of a detergent surfactant and from 0-90% by weight of a builder, characterised in that it further comprises from 0.005-100 LU/mg of a lipolytic enzyme.
2. A process according to claim 1, characterised in that the lipase is a bacterial lipase which shows a positive immunological cross-reaction with the antibody of the lipase, produced by Chromobacter viscosum var. lipolyticum NRRL B 3673.
3. A process according to claim 2, characterised in that the lipase is producible by the microorganism selected from the group consisting of Pseudomonas fluorescens, Pseudomonas fragi, Pseudomonas nitroreducens var. lipolyticum, Pseudomonas cepacia, Pseudomonas gladioli and Chromobacter viscosum.
4. A machine dishwashing process according to any of claims 1-3, characterised in that there is used a composition containing from 20-60% by weight of an alkali metal silicate, from 1-15% by weight of a peroxy type bleaching agent and from 0.5-5% by weight of a low-foaming nonionic detergent.
5. A machine rinsing process according to any of claims 1-3, characterised in that there is used a composition containing, in an aqueous medium, from 0.5-10% by weight of a nonionic surfactant, from 5-25% by weight of an organic or inorganic acid, and from 1-5% by weight of a hydrotrope.

Revendications

1. Procédé pour le lavage ou le rinçage de la vaisselle en machine, comprenant le traitement de la vaisselle avec un liquide aqueux dans lequel est dissoute ou dispersée une quantité efficace d'une

composition enzymatique pour le lavage ou le rinçage de la vaisselle contenant de 0,5 à 10% en poids d'un tensioactif détergent et de 0 à 90% en poids d'un adjuvant, caractérisée en ce qu'elle contient en outre de 0,005 à 100 UL/mg d'enzyme lipolytique.

- 5 2. Procédé selon la revendication 1, caractérisé en ce que la lipase est une lipase bactérienne qui présente une réaction croisée immunologique positive avec l'anticorps dirigé contre la lipase produite par Chromobacter viscosum var. lipolyticum NRRL B 3673.
- 10 3. Procédé selon la revendication 2, caractérisé en ce que la lipase peut être produite par le microorganisme choisi dans le groupe constitué de Pseudomonas fluorescens, Pseudomonas fragi, Pseudomonas nitroreducens var. lipolyticum, Pseudomonas cepacia, Pseudomonas gladioli et Chromobacter viscosum.
- 15 4. Procédé pour lavage de la vaisselle en machine selon l'une quelconque des revendications 1-3, caractérisé en ce qu'on y utilise une composition contenant de 20 à 60% en poids de silicate de métal alcalin, de 1 à 15% en poids d'agent de blanchiment de type peroxy et de 0,5 à 5% en poids de détergent non ionique faiblement moussant.
- 20 5. Procédé de rinçage en machine selon l'une quelconque des revendications 1-3, caractérisé en ce qu'on y utilise une composition contenant, en milieu aqueux, de 0,5 à 10% en poids de tensioactif non ionique, de 5 à 25% en poids d'acide organique ou inorganique et de 1 à 52 en poids d'hydrotrope.

Patentansprüche

- 25 1. Verfahren zum Maschinen-Geschirrwaschen oder -Spülen, das das Behandeln von Geschirr mit einer wässrigen Flüssigkeit, in der eine wirksame Menge einer enzymatischen Geschirrwash- oder Spül-Zusammensetzung, umfassend 0,5-10 Gew.-% eines Detergensoberflächenaktiven Materials und 0-90 Gew.-% eines Builders, gelöst oder dispergiert wurde, umfaßt, dadurch gekennzeichnet, daß es weiterhin 0,005-100 LU(Lipase-Einheiten)/mg eines lipolytischen Enzyms umfaßt.
- 30 2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Lipase eine bakterielle Lipase ist, die eine positive immunologische Kreuzreaktion mit dem Antikörper der Lipase, die durch Chromobacter viscosum var. lipolyticum NRRL B 3673 gebildet wird, zeigt.
- 35 3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß die Lipase durch den Mikroorganismus, ausgewählt aus der Gruppe bestehend aus Pseudomonas fluorescens, Pseudomonas fragi, Pseudomonas nitroreducens var. lipolyticum, Pseudomonas cepacia, Pseudomonas gladioli und Chromobacter viscosum, gebildet werden kann.
- 40 4. Maschinen-Geschirrwash-Verfahren nach einem der Ansprüche 1-3, dadurch gekennzeichnet, daß eine Zusammensetzung verwendet wird, die 20-60 Gew.-% eines Alkalimetallsilikats, 1-15 Gew.-% eines Bleichmittels vom Peroxytyp und 0,5-5 Gew.-% eines schwachschäumenden nicht-ionischen Detergens enthält.
- 45 5. Maschinen-Spülverfahren nach einem der Ansprüche 1-3, dadurch gekennzeichnet, daß eine Zusammensetzung verwendet wird, die in einem wässrigen Medium 0,5-10 Gew.-% eines nicht-ionischen oberflächenaktiven Materials, 5-25 Gew.-% einer organischen oder anorganischen Säure und 1-5 Gew.-% eines Hydrotops enthält.

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